



Xanthan-g-poly(acrylamide): Microwave-assisted synthesis, characterization and *in vitro* release behavior

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ABSTRACT

Xanthan-g-poly(acrylamide) was synthesized employing microwave-assisted and ceric-induced graft copolymerization, and was characterized by FT-IR, DSC, XRD and SEM studies. Matrix tablets of diclofenac sodium were formulated using graft copolymer as the matrix by direct compression technique. Release behavior of the graft copolymer was evaluated using USP type-II dissolution apparatus in 900 ml of phosphate buffer (pH 6.8), maintained at 37 °C and at 50 rpm. Microwave-assisted grafting provided graft copolymer with higher % grafting in a shorter time in comparison to the ceric-induced grafting. The % grafting was found to increase with the increase in the power of microwave and/or time of exposure. The matrix tablets were found to release the drug by zero-order kinetics, and the faster release of drug was observed from the graft copolymer matrix as compared to the xanthan gum matrix. It was observed that grafting reduces the swelling, but increases the erosion of xanthan gum.

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1. Introduction

Natural polymers are preferred over synthetic polymers because of their biodegradability, low cost, easy availability and non-toxicity (Bhardwaj, Kanwar, Lal, & Gupta, 2000). However, they also possess certain drawbacks like uncontrolled hydration, microbial contamination, and drop in viscosity during storage etc. The properties of these natural polymers can be modified by hybridization with synthetic polymers. The chemical combination of natural and synthetic polymers yields new materials which could have desirable properties. The properties of natural polymer have earlier been easily modified by graft copolymerization and formation of interpenetrating network hydrogels with vinyl monomers. One of the natural polymers that have evinced a great interest of researchers is xanthan gum. Xanthan gum has been used widely in pharmaceutical and cosmetic industry as suspending agent, emulsifying agent etc. (Katzbauer, 1998; Talukdar & Kinget, 1995).

In earlier studies graft copolymers of xanthan gum and vinyl monomer have been synthesized and evaluated as controlled release agent (Behari, Pandey, Kumar, & Taunk, 2001; Mundargi, Patil, & Aminabhavi, 2007). Conventionally graft copolymerization is carried out by grafting vinyl monomers onto natural gum using redox initiator (Ei-Tahlawy, Ei-Rafie, & Aly, 2006; Joshi & Sinha, 2007; Margutti et al., 2002; Mishra, Clark, & Pal, 2008; Silva da,

Paula de, & Feitosa, 2007; Trivedi, Kalia, Patel, & Trivedi, 2005; Zohuriaan-Mehr, Motazedi, Kabiri, & Ershad-Langroudi, 2005). The main constraint of graft copolymerization is the formation of concurrent homopolymer resulting in low grafting yield. Apart from the redox initiator-induced graft copolymerization, microwave-assisted graft copolymerization has also been employed. The microwave irradiation is an efficient method which results in rapid transfer of energy in the bulk of the reaction mixture. The microwave-assisted graft copolymerization requires a very short reaction time and proceeds even in the absence of any redox initiator (Singh, Sethi, Tewari, Srivastava, & Sanghi, 2003). In the present study, the graft copolymerization of acrylamide on to xanthan gum was carried out using redox-initiator induced and microwave-assisted graft copolymerization. The acrylamide grafted xanthan gum so prepared, was characterized by FT-IR, XRD, SEM, and DSC studies. The graft copolymer was evaluated for modification of release rate by preparing the matrix tablets of diclofenac sodium.

2. Experimental

2.1. Materials

Xanthan gum (XANTURAL-75, C.P. Kelco, UK) was gifted by Burzin Leons Argenturn Pvt Ltd. (Mumbai, India). Diclofenac sodium (Purity 98.58%) was obtained as gift sample from Dabur Research Foundation (Ghaziabad, India). Acrylamide and ammonium persulphate were procured from Sisco Research Laboratory, (Mumbai, India), and acrylamide used in the study was recrystallized from

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methanol twice, followed by drying at 40 °C in vacuum oven. All other chemicals used were of reagent grade and were used as such.

2.2. Preparation of xanthan-g-poly(acrylamide)

Xanthan-g-poly(acrylamide) was prepared employing microwave-assisted grafting and ceric-induced grafting. Microwave assisted grafting of acrylamide on xanthan gum was done using the method reported earlier (Singh, Tiwari, Tripathi, & Sanghi, 2004). Briefly, xanthan gum (0.2 gm) was dissolved in distilled water by stirring overnight. To this acrylamide (0.568 gm) was added and stirred to dissolve. For grafting with redox-initiator, ammonium persulphate (5.7 mg) was added to this above solution. The solution so obtained was irradiated by microwave in domestic microwave oven (2300 ET-B, Bajaj Electricals Ltd., Mumbai, India) for different times and different powers to prepare various batches of grafted gum. The grafted xanthan gum was treated with acetone, and washed with mixture of methanol and water to remove unreacted monomer and ammonium persulphate. The grafted gum was then dried in an oven at 40 °C for 24 h.

Synthesis of xanthan-g-poly(acrylamide) was also carried out using free radical initiated polymerization employing ceric ammonium nitrate as an initiator (Mundargi et al., 2007). Briefly, 0.2 gm of xanthan gum was dispersed in 20 ml of distilled water and dissolved by constant stirring overnight in a 100 ml round bottom flask. Acrylamide (0.56 gm) was added to the xanthan gum solution and stirred for 1 h. A solution of ceric ammonium nitrate in water equivalent to (0.025 mmol) was added to the above solution. The polymerization was carried out at 60 °C under constant stirring in nitrogen atmosphere for 4 h. At the end of 4 h, the reaction mixture was cooled and added into excess of acetone. Graft copolymer solid so obtained was washed with methanol: water (80:20) mixture to remove the unreacted monomer and reagent followed by drying in vacuum oven at 40 °C to a constant weight. The % grafting, % grafting efficiency, % conversion, % add on and % homopolymer, was calculated using the following equations (Athawale & Lele, 1998).

$$\% \text{ Grafting } (\%G) = \frac{(W_1 - W_0)}{W_0} * 100 \quad (1)$$

$$\% \text{ Grafting Efficiency } (\%GE) = \frac{(W_1 - W_0)}{W_0} * 100 \quad (2)$$

$$\% \text{ Conversion } (\%C) = \frac{W_1}{W_2} * 100 \quad (3)$$

$$\% \text{ Add on } (\%A) = \frac{(W_1 - W_0)}{W_0} * 100 \quad (4)$$

$$\% \text{ Homopolymer} = 100 - \%GE \quad (5)$$

where, W_0 weight of xanthan gum, W_1 weight of graft copolymer, and W_2 weight of acrylamide

2.3. Characterization of grafted gum

The xanthan-g-poly(acrylamide) was characterized by FT-IR spectroscopy, differential scanning calorimetry, X-ray diffractometry and scanning electron microscopy.

2.3.1. FT-IR spectroscopy

The samples were subjected to FT-IR spectroscopy in a Fourier-transform infrared spectrophotometer (Perkin-Elmer, USA) in range of (4000–500 cm^{-1}) as KBr pellet.

2.3.2. Differential scanning calorimetry

Differential scanning calorimetric thermogram of xanthan gum, acrylamide, xanthan-g-poly(acrylamide) and physical mixture of acrylamide and xanthan gum was recorded using differential

scanning calorimeter (Q10, TA Systems, USA) in the temperature range of (40–250 °C) at a heating rate of 10 °C per minute in nitrogen atmosphere.

2.3.3. X-ray diffractometry

X-ray diffractogram of xanthan gum, acrylamide and xanthan-g-poly(acrylamide) samples were recorded employing X-ray diffractometer (XpertPRO, Panalytical, Germany) using copper $K\alpha$ radiation generated at 40 kV and 35 mA in the differential angle range of 3–70° 2θ using an X-ray diffractometer.

2.3.4. Scanning electron microscopy

Scanning electron micrograph of xanthan gum, acrylamide, and xanthan-g-poly(acrylamide) particles were taken using a SEM (268D, Fei-Philips Morgagni). These were coated with gold and mounted in a sample holder. The photomicrograph of sample was taken at an accelerating voltage at 15 kV at different magnifications.

2.4. Preparation of matrix tablets of diclofenac sodium

Matrix tablets of diclofenac sodium were prepared employing graft copolymer as the matrix. The required quantity of diclofenac sodium (75 mg) was blended with xanthan gum or grafted xanthan gum (75 mg) and the magnesium stearate (1.5 mg) as lubricant. The dry blend so obtained was directly compressed using 8 mm biconvex punches and dies in a single station hand-operated, tableting machine (R&D model, Konark Instruments, Ambala, India).

2.5. Evaluation of tablets

The matrix tablets of diclofenac sodium were evaluated for thickness, weight variation, hardness, friability, content uniformity, and *in vitro* release.

2.5.1. Thickness and diameter

Thickness of randomly selected twenty tablets was determined using vernier caliper (Aerospace, China). Values are reported as mean \pm SD.

2.5.2. Weight variation

The weight of 20 tablets of each batch was measured individually using electronic balance (AND, Japan) and standard deviation was calculated.

2.5.3. Hardness

The hardness of six tablets of each batch was measured using Monsanto hardness tester (Macro Scientific, New Delhi).

2.5.4. Friability

To determine the friability, six tablets of each batch were weighed and placed in a friabilator (Campbell Electronics, Mumbai, India). The tablets were rotated for 4 min at 25 rpm. The tablets were then dedusted and collected, and reweighed. The friability was calculated as the percentage weight loss.

2.5.5. Uniformity of content

The content uniformity of the prepared batches of tablets was determined in triplicate, by powdering six tablets from each batch in a pestle mortar and extracting the powder equivalent to 100 mg of diclofenac sodium with 100 ml of phosphate buffer (pH 6.8) by sonication (for 10 min). The aqueous suspension so obtained, was filtered employing 0.45 μ syringe filter and content of diclofenac sodium in the solution was determined by measuring absorbance at 276 nm after suitable dilution.

2.5.6. In vitro release study

The *in vitro* release study of diclofenac sodium from the prepared batches of tablets was conducted in triplicate using six tablets from each batch, employing USP type II dissolution apparatus (TDT-08L, Electrolab, India). Dissolution media comprised of 900 ml phosphate buffer (pH 6.8) till 16 h, maintained at 37.0 ± 0.5 °C and 50 rpm. An aliquot of 5 ml sample was withdrawn and replaced with another 5 ml of fresh dissolution medium at various time intervals. The contents of diclofenac sodium in sample were determined by measuring the absorbance at 276 nm in UV–Visible Spectrophotometer (Carry 5000, Varian Australia).

2.6. Modeling and release kinetics

To determine the order and mechanism of diclofenac sodium release from matrix tablets, the release rate data was fitted to zero-order, first-order and Higuchi square-root equation (Costa & Loba, 2001). The value of k (the release rate constant) for different models was determined. However, these equations fail to explain the drug release mechanism from matrices that undergo swelling and/or erosion during dissolution. Therefore, the dissolution data was fitted to the Korsmeyer–Peppas equation, which is often used to describe the drug release mechanism from polymeric system.

$$\log(M_t/M_f) = \log k + n \log t \quad (6)$$

where M_t is the fraction of drug release at time t , M_f is the amount of drug release after infinite time, and k is the release rate constant incorporating structural and geometric characteristics of the tablets and n is the diffusion exponent indicative of the mechanism of the release mechanism. To determine the release exponent, n for different batches of matrix tablets, the log value of percentage drug dissolved was plotted against log time for each batch, according to Eq. (6). For determination of exponent n , only the initial portion of release curve ($M_t/M_f < 0.6$) was used. A value of $n = 0.45$ indicates Fickian (case I) release; the rate of drug release is much less than that of polymer relaxation (erosion). Thus, the release of drug is primarily by diffusion through the matrix. A value of n ($0.45 < n < 0.89$) indicates non-Fickian (anomalous) release, the release of the drug occurs by combined effect of drug diffusion and polymer relaxation. The value of $n > 0.89$ indicates super case II type of release. Case II generally refers to the erosion of the polymeric matrix (Ravi, Ganga, & Saha, 2007).

2.7. Swelling and erosion behavior

Swelling and erosion studies were conducted using the method reported earlier (Bashar, Taani, & Tashtoush, 2003). Matrix tablet was introduced into dissolution apparatus under the standard conditions as specified earlier in, *in vitro* drug release study. The tablets were removed using a small basket and swollen weight of each tablet was determined. To assess the matrix erosion, swollen tablets were kept in a vacuum oven at 40 °C for 48 h. Then the tablets were removed and weighed. The % swelling and % erosion were calculated according to the following formula, where S is the weight of matrix and R is the weight of eroded matrix. T is the initial weight of tablet.

$$\% \text{ Swelling} = S/R * 100 \quad (7)$$

$$\% \text{ Erosion} = (T - R)/T * 100 \quad (8)$$

3. Results and discussion

3.1. Effect of grafting conditions on grafting parameters

Chemical modification of xanthan gum was carried out by the graft polymerization of acrylamide onto xanthan gum. Many meth-

Table 1

Grafting parameters from various batches of microwave-assisted and ceric-induced grafting.

Microwave		Grafting (%G)	Grafting efficiency (%GE)	Conversion (%C)	Add on (%A)	Homopolymer (%H)
Power	Time (s)					
40	40	12.76	4.49	39.70	11.32	95.50
40	100	120.30	42.35	77.57	54.60	57.64
60	40	110.33	38.84	74.06	52.45	61.15
60	100	129.86	45.72	80.93	56.49	54.27
80	40	126.36	44.49	79.70	55.82	55.50
80	100	149.63	52.68	87.89	59.94	47.31
100	40	87.15	30.68	65.89	46.56	69.31
100	100	190.28	67.00	102.21	65.55	33.00
Ceric-induced grafting		62.87	22.33	57.35	38.60	77.86

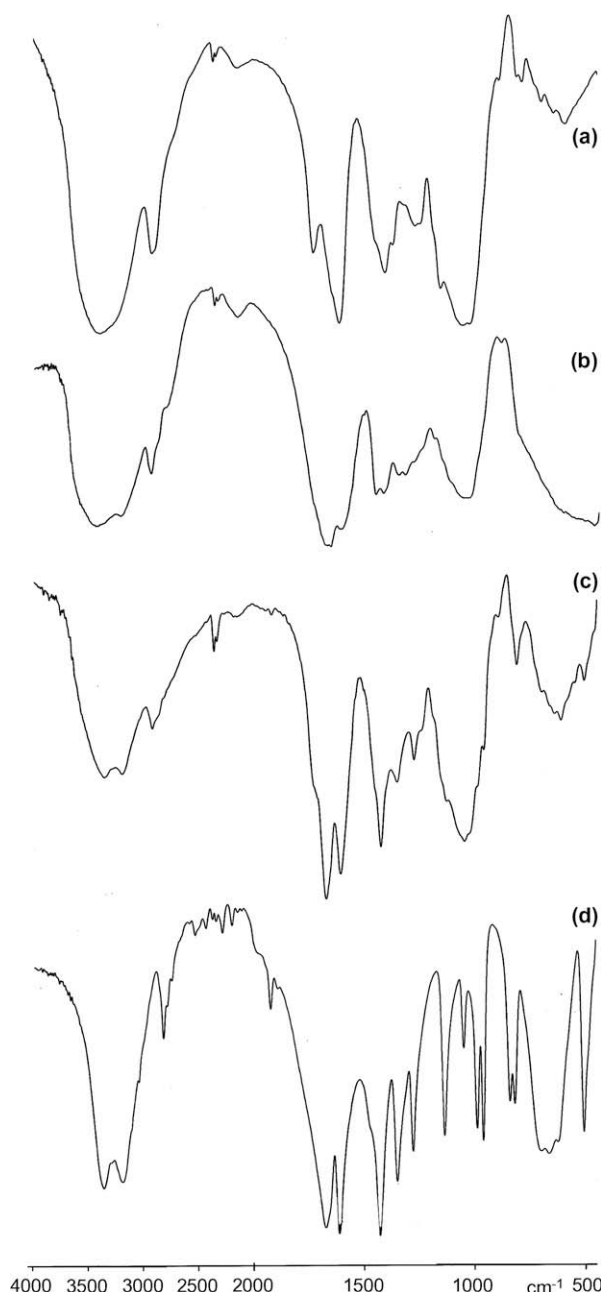


Fig. 1. FT-IR spectra of (a) xanthan gum, (b) acrylamide, (c) microwave-assisted xanthan-g-poly(acrylamide), (d) ceric-induced xanthan-g-poly(acrylamide).

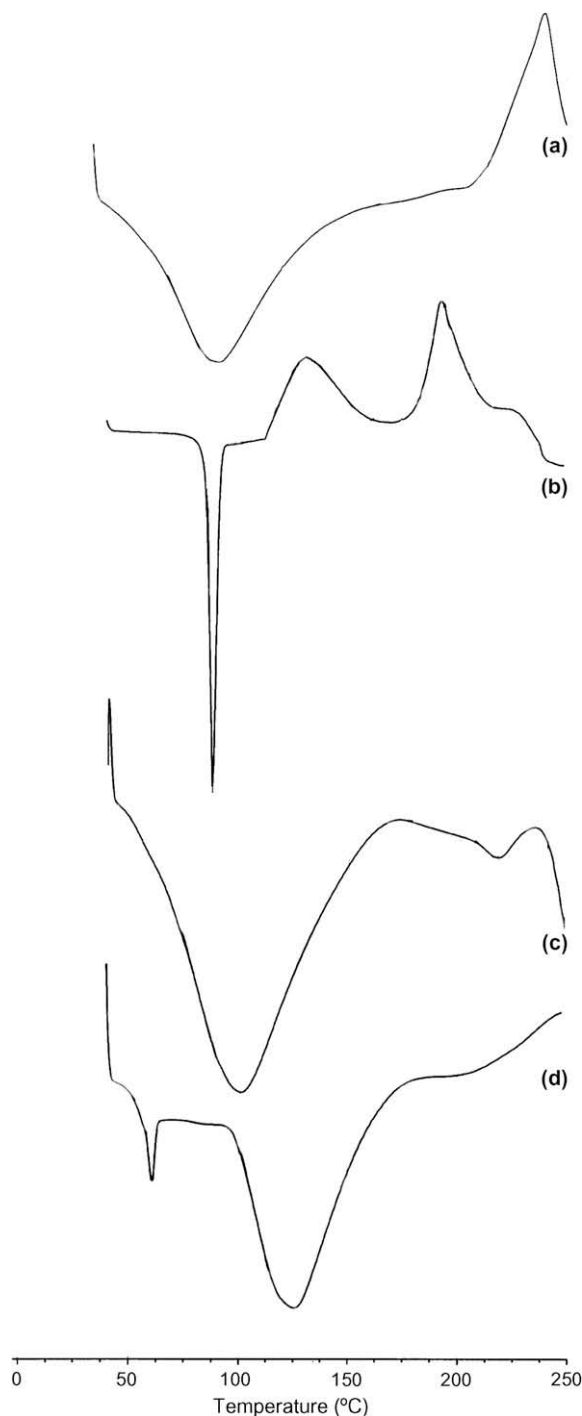


Fig. 2. DSC curve of (a) xanthan gum, (b) acrylamide, (c) microwave-assisted xanthan-g-poly(acrylamide), (d) ceric-induced xanthan-g-poly(acrylamide).

ods of graft copolymerization of acrylamide or another monomer onto natural gums have been reported. Ceric ammonium nitrate is usually employed for initiating free radical induced graft copolymerization. In an earlier study, acrylamide grafted xanthan gum was prepared using ceric-induced free radical graft copolymerization (Mundargi et al., 2007). Recently, microwave-assisted graft copolymerization of acrylate monomer on to guar gum (Singh et al., 2004), chitosan (Singh, Tiwari, Tripathi, & Sanghi, 2006), starch (Huang & Chen, 1999) and artemisia gum (Zhang, Zhang, Yuan, & Wang, 2007) have been reported. In the present study, the microwave-assisted graft copolymerization of acrylamide on

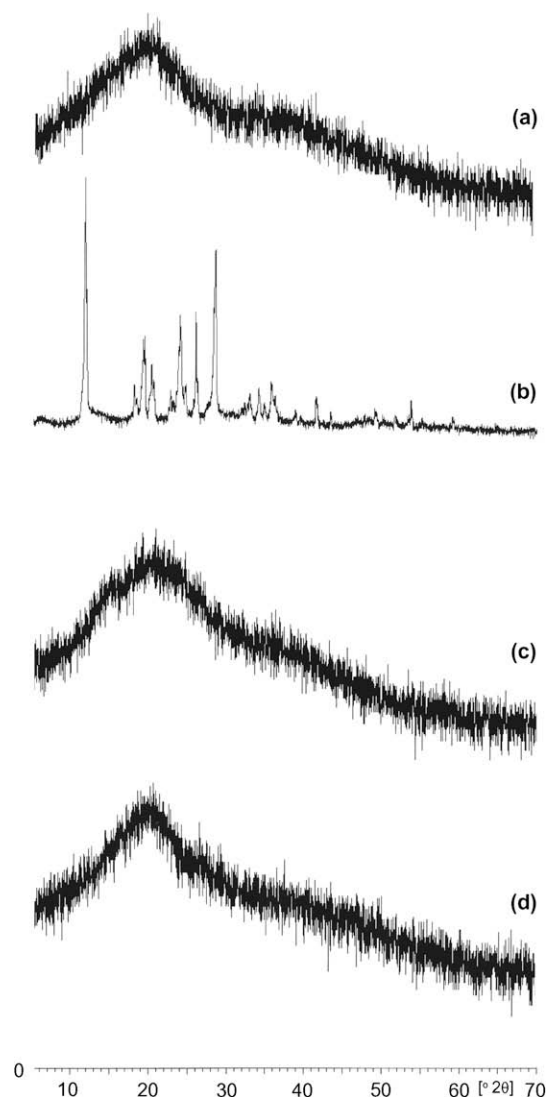


Fig. 3. X-ray diffractogram of (a) xanthan gum, (b) acrylamide, (c) microwave-assisted xanthan-g-poly(acrylamide), (d) ceric-induced xanthan-g-poly(acrylamide).

xanthan gum has been explored and compared with the conventional ceric-induced graft copolymerization. Table 1 presents the result of microwave-assisted graft copolymerization and ceric-induced graft copolymerization of acrylamide on xanthan gum (carried out under various combinations of microwave power and time).

It can be observed from the results that microwave assisted graft copolymerization provided higher % grafting, % grafting efficiency and % conversion as compared to the ceric-induced graft co-polymerization. Further, it can be observed that as we go on increasing the power and time of microwave irradiation there occurs a corresponding increase in % grafting, % grafting efficiency, % conversion, % add on and reduction in % homopolymer formation.

3.2. Characterization of xanthan-g-poly(acrylamide)

Acrylamide grafted xanthan gum was characterized by FT-IR, DSC and XRD study. Fig. 1 exhibits the FT-IR spectra of xanthan gum, acrylamide, acrylamide grafted xanthan gum prepared by microwave-assisted grafting and ceric-induced grafting. The IR spectra of xanthan gum showed the broad absorption band at

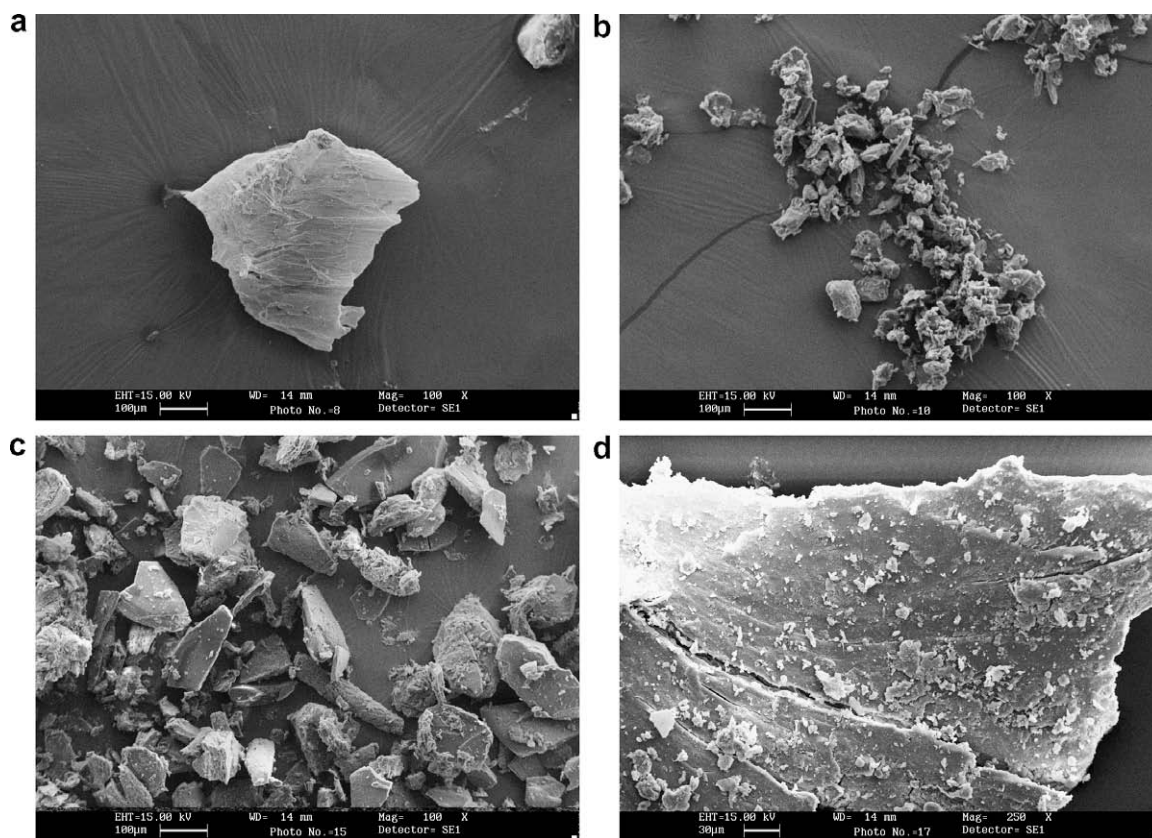


Fig. 4. Scanning electron micrograph of (a) acrylamide, (b) xanthan gum, (c) microwave-assisted xanthan-g-poly(acrylamide), (d) ceric-induced xanthan-g-poly(acrylamide).

3386 cm^{-1} owing to the stretching frequency of the OH group. Absorption band at 2919 cm^{-1} appeared due to CH stretching of alkyl group, and at 1410 cm^{-1} due to CH bending of methyl group. The presence of absorption band at 1733, 1616 cm^{-1} can be attributed to CO stretching of alkyl esters and asymmetric stretching of carboxylate ion. The band at 1060 cm^{-1} appears due to CO stretching of alcohol. The spectra of acrylamide presented absorption bands at 3354 cm^{-1} due to asymmetric NH stretching and at 3180 cm^{-1} due to symmetric NH stretching of NH group. The amide-I band (CO stretching) appear at 1673 cm^{-1} and amide-II (NH bending) appear at 1612 cm^{-1} . The band at 1428 cm^{-1} can be attributed to CN stretching, while the CH stretching appear at 2812 cm^{-1} and CH out of plane bending at 988 cm^{-1} . The broad NH out of plane wagging appears at 818 and 664 cm^{-1} . The IR spectra of chemically grafted xanthan gum showed a broad absorption band at 3358 cm^{-1} due to overlap of OH stretching band of xanthan gum and NH stretching band of acrylamide. The amide-I (CO stretching) occurred at 1675 cm^{-1} and amide-II (NH bending) occurred at 1611 cm^{-1} . The CH stretching band of xanthan gum and acrylamide overlap to give a CH stretching at 2923 cm^{-1} the CO stretching contributed by the xanthan gum appeared at 1051 cm^{-1} . The CN stretching of acrylamide appeared at 1429 cm^{-1} .

The IR spectra of microwave grafted xanthan gum showed the broad absorption band at 3420 cm^{-1} due to overlap of OH stretching of xanthan gum and NH stretching of acrylamide. The CO stretching of carboxylate overlaps with amide-I to give broad absorption band at 1654 cm^{-1} . The strong absorption band at 2928 cm^{-1} are attributed to overlap of CH stretching of xanthan gum and acrylamide. The OH stretching of alcohol contributed by xanthan gum occurs at 1038 cm^{-1} . The CH bending of methyl group of xanthan gum overlaps with the CN stretching of acrylamide to give a broad absorption band at 1453 cm^{-1} .

Fig. 2 shows the DSC curve of xanthan gum, acrylamide and acrylamide grafted xanthan gum prepared both by using microwave-assisted and ceric-induced graft co-polymerization. The thermogram of xanthan gum showed a broad endotherm at 108.91 $^{\circ}\text{C}$ with heat of fusion of 392.1 J/g. The thermogram of acrylamide showed the sharp endothermic peak at 87.5 $^{\circ}\text{C}$ with heat of fusion of 199.7 J/g. DSC curve of acrylamide also showed two exotherms at 131.4 $^{\circ}\text{C}$ with heat flow of 291.8 J/g and at 204.1 $^{\circ}\text{C}$ with heat flow of 50.28 J/g.

The DSC curve of acrylamide grafted xanthan gum prepared by microwave irradiation showed the broad endotherm at 105 $^{\circ}\text{C}$ with heat of fusion 181.6 J/g and another endotherm at 270.16 $^{\circ}\text{C}$ with heat of fusion of 8.71 J/g, while the thermogram of acrylamide grafted xanthan gum prepared by ceric-induced polymerization showed two endothermic peaks at 60.6 and 160.2 $^{\circ}\text{C}$ with heat of fusion of 3.83 and 105.9 J/g, respectively.

Fig. 3 displays the X-ray diffractogram of xanthan gum, acrylamide, and acrylamide grafted xanthan gum prepared both by microwave-assisted and ceric-induced graft co-polymerization. The XRD spectra showed the amorphous nature of xanthan gum, microwave-assisted acrylamide grafted xanthan gum, and ceric-induced acrylamide grafted xanthan gum, while the diffractogram of acrylamide showed the crystalline nature of acrylamide with the characteristic peak of acrylamide appearing at 12, 18, 19, 20, 23, 26 and 29 $^{\circ}2\theta$.

Fig. 4 shows the scanning electron micrographs of acrylamide, xanthan gum, microwave-assisted acrylamide grafted xanthan gum and ceric-induced acrylamide grafted xanthan gum, respectively. The acrylamide particles are polyhedral in shape, while the xanthan gum particles are fibrous in nature. The SEM images of grafted copolymer show that the grafting of acrylamide onto xanthan gum brings about the change in the shape and size of the xanthan gum particles. Further, it can be observed that the

Table 2
Physical characteristics of tablets.

Batch	Thickness (mm) ^a	Diameter (mm) ^a	Average weight (mg) ^a	Hardness (kg/cm ²) ^b	Assay (%) ^b	Friability (% w/w) ^b
F1	2.90 ± 0.20	8.10 ± 0.01	151.5 ± 0.02	4.1 ± 0.80	97.4 ± 0.2	0.42 ± 0.18
F2	3.01 ± 0.02	8.08 ± 0.10	151.0 ± 0.10	4.4 ± 0.51	98.4 ± 0.5	0.42 ± 0.04
F3	3.10 ± 0.12	8.18 ± 0.03	151.8 ± 0.08	4.2 ± 0.70	98.9 ± 0.6	0.43 ± 0.10
F4	3.12 ± 0.08	8.12 ± 0.04	151.3 ± 0.03	4.1 ± 0.14	97.9 ± 0.2	0.44 ± 0.08
F5	2.94 ± 0.10	8.11 ± 0.01	151.2 ± 0.01	4.3 ± 0.35	99.4 ± 0.4	0.48 ± 0.15
F6	3.02 ± 0.05	8.12 ± 0.11	152.1 ± 0.11	5.0 ± 0.45	98.7 ± 0.5	0.52 ± 0.02

F1, grafted xanthan gum (40 power, 100 s); F2, grafted xanthan gum (60 power, 100 s); F3, grafted xanthan gum (80 power, 100 s); F4, grafted xanthan gum (100 power, 100 s); F5, xanthan gum; F6-ceric-induced acrylamide grafted xanthan gum.

^a Values are mean ± SD (n = 20).

^b Values are mean ± SD (n = 6).

particles of acrylamide grafted xanthan gum are bigger in size than ungrafted xanthan gum.

The results of physical characterization of tablets are presented in Table 2. The physical appearance, hardness, friability, weight variation and drug content of all the formulated matrix tablets were found to be satisfactory and reproducible. Fig. 5 compares the dissolution profile of the diclofenac sodium from the matrix tablets of xanthan gum, acrylamide grafted xanthan gum and commercial diclofenac sodium [Voveran-SR (75 mg)]. It can be observed from results that the commercial diclofenac sodium (Voveran-SR) released 100% of the drug in 10 h, while the matrix tablet formulated using xanthan gum released only 49% of the drug in 16 h. Further, it can be observed that the release rate of drug from matrix tablet formulated with acrylamide grafted xanthan gum was higher than the tablet formulated with ungrafted xanthan gum. Result also showed that among the acrylamide grafted xanthan gum matrix tablets, the release rate increased with increase in % grafting. The tablets of batch F4 which had % grafting of 190% released almost 100% of drug in 12 h, while the tablets of batch F1 with % grafting of 120% released only 76% of the drug in 16 h. The matrix tablets formulated using ceric-induced acryl-

Table 3
Modeling and release kinetics.

Batch	Zero-order		First-order		Higuchi square-root		Peppas		
	R ²	K (%h ⁻¹)	R ²	K (h ⁻¹)	R ²	K (%h ^{-0.5})	R ²	K (%h ⁻ⁿ)	n
F1	0.998	4.81	0.832	0.203	0.945	21.15	0.993	6.27	0.861
F2	0.994	4.84	0.852	0.208	0.920	21.39	0.991	6.54	0.874
F3	0.998	6.07	0.832	0.210	0.942	27.09	0.991	5.46	0.881
F4	0.985	8.03	0.786	0.220	0.954	36.31	0.996	6.81	0.973
F5	0.989	3.09	0.755	0.187	0.982	14.15	0.998	7.58	0.814
F6	0.984	3.75	0.794	0.179	0.975	17.16	0.994	4.41	0.872
Voveran-SR	0.917	6.67	0.706	0.124	0.992	31.87	0.998	1.76	0.650

amide grafted xanthan gum which has 62.87% grafting, released only 60% of the drug in 16 h. Thus, the release rate of the drug from the matrix tablets increased with increase in the grafting of acrylamide onto xanthan gum. The results of release rate study are contrary to the earlier study (Mundargi et al., 2007) which reported decreased release rate of carvedilol and atenolol from the acrylamide grafted xanthan gum matrices as compared with ungrafted xanthan gum matrices. Apart from the nature and concentration of the matrix polymer, release rate also depends on the physico-chemical properties of the drug. This might be one of the reasons for the contradictory results.

Table 3 shows the results of modeling and release kinetics. The result of modeling study revealed that the release of drug from matrix tablets of xanthan gum and acrylamide grafted xanthan gum fitted best into zero-order kinetics, while the release from, commercial diclofenac sodium fitted best into Higuchi square-root kinetics. Further, the value of 'n' (0.45 ≤ n ≤ 0.89), the release exponent of Korsmeyer–Peppas indicated that all batches of tablets, except the tablet of batch F4, released the drug by combination of diffusion through the matrix and matrix erosion i.e. anomalous transport. The tablet of batch F4 which employed copolymer with highest grafting, released the drug by matrix erosion as the value of n = 0.973 (n > 0.89). It can be noted that as the % grafting increased, the value of n also increased. The results of kinetic study are consistent with the swelling and erosion study. The % swelling of the matrix tablets was found to be in the order F5 > F6 > F1 > F2 > F3 > F4 > Voveran-SR, while the % erosion of tablets ranked in the order F4 > F3 > Voveran-SR > F1 > F2 > F6 > F5. It was observed that the matrix tablets formulated employing ungrafted xanthan gum (F5) showed the highest swelling and least erosion, while the tablets formulated using graft copolymer with highest grafting showed the highest erosion.

4. Conclusion

Xanthan-g-poly(acrylamide) was synthesized using microwave-assisted grafting and ceric-induced grafting. The % grafting was found to be higher with microwave-assisted grafting as compared to ceric-induced grafting, and was found to be directly proportional to the microwave power and exposure time. The graft copolymer was evaluated for modification of release rate employing the matrix tablets of diclofenac sodium. The study revealed the faster release of drug from graft copolymer matrix tablets as compared with the ungrafted xanthan gum. The release rate increased with the increase in % grafting, and was found to follow zero-order release kinetics. The swelling of the xanthan gum was found to vary inversely with the % grafting, while erosion varied directly with the % grafting. Thus, microwave-assisted graft co-polymerization can be used as an efficient tool to modify the release properties of xanthan gum by grafting of acrylamide on xanthan gum.

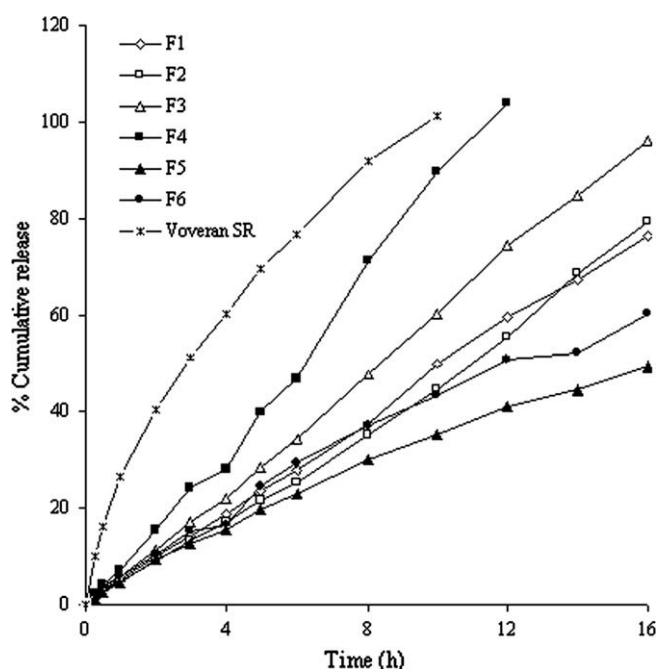


Fig. 5. Comparative *in vitro* release profile of diclofenac sodium from various batches of matrix tablets of xanthan, xanthan-g-poly(acrylamide) and Voveran-SR.

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